How to Use a Synchrotron for IR Microscopy

Infrared spectromicroscopy combines the well-established technique of Fourier transform infrared (FTIR) spectroscopy with a customized microscope. Together they measure infrared spectra from a tiny part of a sample. Most FTIR spectrometer instrumentation companies now offer one or more IR microscopes in their product line.

Conventional mid-infrared sources used in FTIR instruments are thermal emission elements that produce a graybody spectrum from a filament heated to1000 to 2000 K. These elements can be rod-, coil-, or u-shaped, physically large (at least several millimeters), and typically radiate in all directions. The FTIR bench optics collect the light, then collimate and pass it through the scanning interferometer. Next, this modulated light is sent into the IR microscope. The IR microscope objective and condenser optics are reflective and focus the IR light to a small spot on a sample. Finally, the light that the sample reflects or transmits is collected, focused onto a detector, and processed by a computer to produce an infrared spectrum.

The brightness (flux/area) attainable in IR spectromicroscopy is governed primarily by how point-like the source is. The thermal emission sources can be focused down to 75 to 100 µm with an IR microscope. To measure something smaller, you must mask away part of the incoming light, significantly reducing the signal strength. A true point source could be focused to a diffraction-limited spot size, with f/1 optics this is approximately the wavelength of the light. This is where using a synchrotron as an IR source really shines.

In the mid-IR wavelengths—3 to 20 µm—the effective source size for a typical synchrotron light source is nearly diffraction-limited. In other words, it is very close to an ideal point source. This means that in FTIR spectromicroscopy based on synchrotron radiation (SR), the beam is focused to a spot with a diameter ⩽10 µm, smaller than a typical mammalian cell. This provides hundreds of times the brightness of conventional IR sources.

The sample can be positioned using a computer-controlled x-y stage with 1-µm accuracy, allowing mapping measurements of FTIR spectra as a function of x and y position on the sample.

The considerably higher brightness available at synchrotron IR spectromicroscopy facilities enables a multitude of new scientific applications where size matters.

Environmental scientists want to study chemical states and reactions on heterogeneous soil and rock surfaces. By focusing the SR-IR beam on one soil grain, users can probe the interactions occurring on localized regions of these surfaces. Additionally, because of the high signal-to-noise ratio available, one can measure dilute concentrations that are realistic and relevant to what is in the environment. One recently published article followed the biodegradation of chromium (VI), a heavy metal toxic pollutant, to chromium (III), which is relatively benign, by a tiny colony of indigenous bacteria.

IR light has the additional property of being too low in energy to harm biomolecules the way visible laser, ultraviolet, and x-ray probes do. This, combined with its small beam size, allows scientists to investigate individual living human cells and monitor their reactions to stimuli, such as drug uptake, other chemicals, or exposure to radiation. By carefully analyzing the acquired SR-FTIR spectra, one can learn about damage and repair mechanisms in living cells—key for understanding how diseases start and spread, as well as how aging and death occur. For example, finding spectral markers that change when a cell is cancerous could lead to new medical diagnostic tools, such as a fiber optic probe that could determine if a tumor is malignant or has spread.

Other applications of SR-FTIR spectromicroscopy include studying conformational changes in protein microcrystals, analyzing tiny forensic evidence samples, investigating surface chemistry in corrosion or battery cathodes and anodes, analyzing individual hairs before and after treatments, locating semiconductor defects, and probing unusual states of liquids with microjets.

The exciting applications of SR-FTIR spectromicroscopy span almost every scientific discipline and have driven a number of beamlines to be built around the world. Currently, such facilities are in use at Brookhaven National Laboratory, Upton, N.Y.; Lawrence Berkeley National Laboratory, Berkeley, Calif.; the National Institute of Standards and Technology, Gaithersburg, Md.; the Synchrotron Radiation Center, Madison, Wis.; and the Laboratoire pour l'Utilisation du Rayonnement Electromagnétique, Orsay, France.

New IR beamlines are also being developed at the synchrotron facilities in Daresbury, UK; Lund, Sweden; Dortmund, Germany; and Hefei, China. They are also planned for the new Canadian Light Source, Saskatoon, Saskatchewan; BESSY II (Berliner Elektronenspeicherring-Gesellschaft für Synchrotronstrahlung mbH), Berlin; ANKA (Angströmquelle Karlsruhe GmbH), Karlsruhe, Germany; the Swiss Light Source, Villigen, Switzerland; and SESAME (Synchrotron-light for Experimental Science and Applications in the Middle East).

All of these synchrotron light sources have mechanisms for outside scientists to come and perform research on the facilities. To solve a research problem that requires mapping or individual measurements on the 10-µm scale, contact the facility of your choice. ***—Michael C. Martin***

Martin is a scientist at the Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, Calif.

Links to the synchrotron facilities mentioned are available at www.rdmag.com/basics/basics.htm.